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IS 1448-103 (1981): Methods of test for petroleum and its products, Part 103: Barium, calcium, phosphorous and zinc in lubricating oils by direct reading emission spectrographic method [PCD 1: Methods of Measurement and Test for Petroleum, Petroleum Products and Lubricants]

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IS : 1448 [P : 103] - 1981
Adopted : 11 February 1981

(Reaffirmed 2003)

Indian Standard

METHODS OF TEST FOR PETROLEUM AND ITS PRODUCTS

[P : 103]

BARIUM, CALCIUM, PHOSPHORUS AND ZINC IN LUBRICATING OILS BY DIRECT READING EMISSION SPECTROGRAPHIC METHOD

UDC 621·892 : 543·423·083·1

Adapted from Institute of Petroleum, UK Standard No. IP 187/66 (Reapproved 1975)

1. SCOPE

1.1 This method is intended for the routine determination of barium, calcium, phosphorus, and zinc in unused lubricating oils and is suitable for the concentration range 0·01 to 0·5 percent m/m of the elements, provided that the nature of all the additives present is known. Spectral interferences due to impurities in the electrodes or samples are assumed to be absent.

2. OUTLINE OF THE METHOD

2.1 The sample is sparked between graphite electrodes, the upper electrode being a rod having a conical end, while the lower electrode is a rotating disc, the lower edge of which dips into the oil and feeds a continuous film into the spark gap. A direct reading spectrograph is used to measure the emission of selected radiations, and the measurements are converted to element concentrations by reference to appropriate calibration curves.

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July 1981

2.2 Any interference due to an inter-element effect or additive type is eliminated by employing standards containing the same additives as the sample, whether these are to be determined or not. The effect of physical properties of the oils, such as viscosity, are similarly eliminated by preparing standards in the same type of base oil as the sample.

3. APPARATUS

3.1 Direct Reading Emission Spectrograph — equipped with a rotating disc electrode assembly and provided with sample containers. The instrument should be capable of measuring radiation at wavelengths of 233·5 nm (Ba), 315·9 nm, 317·9 nm, 422·6 nm (Ca), 253·5 nm (P) and 213·8 nm (Zn). For zinc wavelength 334·5 nm (Zn) may also be used.

NOTE — Instruments using a measurement of background intensity at or near 293·2 nm for internal standardization may suffer from vanadium, iron, and manganese interference at this wavelength. Iron also interferes with some analytical lines, particularly phosphorus at 253·5 nm.

3.1.1 Provision for maintaining the spectrograph at a constant temperature is preferred.

3.2 Source Unit — capable of giving a 15 kV simple condensed spark with added inductance.

3.3 Cutter — for shaping the upper electrodes.

4. MATERIALS

4.1 Graphite Rods — of approximately 6·5 mm diameter, spectrographic quality, and befitting the particular instrumental setup.

4.2 Graphite Discs — of approximately 13 mm diameter, 3 mm thickness, spectrographic quality, befitting the particular instrumental setup, and drilled centrally to fit the spindle of the electrode assembly.

4.3 Standards — additives of known composition appropriate to the samples being analysed, and base oils of similar viscosity characteristics to those of the samples. Alternatively, chemically analysed lubricating oil samples (*see* Note) of similar compositions to the samples under test, and covering a suitable concentration range may be used.

NOTE — The accuracy of the chemical methods employed shall be at least equivalent to that given by the current IS methods for the individual elements.

5. PREPARATION OF APPARATUS

5.1 Set the source unit to give a 15 kV condensed spark discharge and an inductance which gives adequate sensitivity for all elements.

5.2 Align the spectrograph to give optimum performance and set the controls as follows.

5.2.1 *Disc Rotation Speed* — 5 rev/min.

5.2.2 *Pre-spark Time* — that required to complete at least one revolution of the disc.

5.2.3 *Exposure Time* — controlled by the internal standard (background) channel and should be of sufficient duration to ensure good repeatability. Times of 20 to 40 seconds have been found satisfactory.

6. CALIBRATION

6.1 Preparation of Standards — Prepare sets of standards covering the anticipated concentration range for each type of lubricating oil to be tested, by either:

a) using additives of known composition and a base oil appropriate to the samples; or

b) using chemically-analysed lubricating oils similar to the samples (*see* **4.3**). Samples of lower concentration than a single master standard may be obtained by accurate dilution with a suitable base oil.

6.1.1 A minimum of five standards shall be prepared. Heating up to 60°C is permissible to facilitate blending.

6.2 Preparation of Calibration Curves

6.2.1 Place a graphite disc in position on the spindle.

6.2.2 Shape one end of a suitable length of graphite rod to a 90 degree included angle cone with rounded tip. Place the electrode in the upper clamp and set the gap to 3 mm.

6.2.3 Fill one of the sample containers with the standard containing the highest concentration of the elements and place on the stand below the rotating disc. Adjust the position of the stand so that the rim of the disc just dips below the surface of the oil.

6.2.4 Initiate the spark discharge, and at the end of the exposure, adjust the instrument to give approximately 75 percent full scale deflection on this standard. Record the duration of the exposure and the meter readings on the 'Auto' and element channels.

6.2.5 Without any further adjustment of the instrument repeat the procedure twice from **6.2.1** for this standard and subsequently in triplicate for the other standards.

6.2.6 Average the meter readings for the triplicate sparkings for each element on each standard and plot against element concentration on linear graph paper.

7. PROCEDURE

7.1 Two methods are given. Method A is convenient where the analysis follows immediately on calibration; otherwise Method B shall be used.

7.2 Method A

7.2.1 Maintaining the conditions employed in the calibration, but using the sample to be tested, carry out the operation detailed in **6.2.1** to **6.2.3**. Spark the sample in triplicate and record the meter readings for each element present.

7.3 Method B

7.3.1 Carry out operations detailed in **6.2.1** to **6.2.4**.

7.3.2 Without any further adjustment of the instrument repeat this procedure a further three times.

7.3.3 For each element calculate the difference between the mean meter readings of the four sparkings and the value of the corresponding meter readings for the element concentrations given by the calibration curve. These differences are added to, or subtracted from, the meter readings obtained on the last of the replicate sparkings by varying the fine sensitivity controls.

7.3.4 Proceed as in Method A (*see* **7.2**).

8. CALCULATION AND REPORTING

8.1 For each element, calculate the arithmetic mean of the meter readings from the triplicate sparkings, and using the appropriate calibration curves, determine the concentration of the elements present in the sample.

8.2 Report the results calculated to the nearest 0·01 percent m/m at concentrations above 0·1 percent m/m and to the nearest 0·001 percent m/m at concentrations below 0·1 percent m/m, as the barium, calcium, phosphorus and zinc contents.

9. PRECISION

9.1 The following criteria should be used for judging the acceptability of results (95 percent confidence).

9.1.1 *Repeatability* — Duplicate results by the same operator should be considered suspect if they differ by more than the following amounts, calculated as a percentage of the lowest:

	Ba	Ca	P	Zn
		Percent		
Method A	9	12	14	13
Method B	11	—	10	16

9.1.2 *Reproducibility* — The results submitted by each of two laboratories should not be considered suspect unless the two results differ by more than the following amounts, calculated as a percentage of the lowest:

	Ba	Ca	P	Zn
		Percent		
Method A	18	24	21	23
Method B	23	—	39	29

9.2 Because of insufficient data, no precision figures have been quoted for calcium by Method B.

NOTE — Figures for precision for zinc is only for 213·8 nm line. Precision data for 334·5 nm line will be evolved.

Printed at Britannia Calendar Mfg Co, Calcutta, India